
घरेलू धूलाई के लिए अपमार्जक
पाउडर — विशिष्टि
(पाँचवां पुनरीक्षण)

Household Laundry Detergent
Powders — Specification
(Fifth Revision)

ICS 71.100.40

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FOREWORD

This Indian Standard (Fifth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Soaps and other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

Synthetic detergents produced in the country are mainly of the alkyl aryl type, such as sodium salts of alkyl benzene sulphonic acid for which a separate Indian Standard IS 9985 Specification for sodium alkyl benzene sulphonate, technical is available.

This Indian Standard was originally published in 1968 and it covered primarily the spray dried powders produced by large scale sector. It was revised for the first time in 1978 incorporating another grade of the material produced by dry mixing of the ingredients, such powders have higher bulk density as compared to those produced by spray drying process. Thereafter, the standard was revised in 1982 incorporating two additional grades of the material in accordance with prevailing manufacturing practice and for the third time in 1991. During the third revision the requirement for *pH* had been substituted with 'active alkalinity' as it was felt that this characteristic would more explicitly reflect the effect of alkaline materials used in the formulation of detergents and their skin irritation potential from the physico-chemical angle as compared to the requirement for *pH*. The limits for active detergents and sodium tripolyphosphate (STPP) were modified. In the fourth revision performance based requirements incorporated and Grade 3 had been deleted as distinction of product of Grade 3 and Grade 4 for performance like cleaning and ash built up was not possible. Performance requirements like detergency and ash built up had also been incorporated in fourth revisions it matches the performance defined by the current Grades 1, 2 and 3 formulations of the standard and for the modified Grade 3 limit for active ingredient had been changed.

Presence of phosphorous in excess in water body is known to cause eutrophication leading to algal growth, thereby posing risks to aquatic life. Apart from fertilizer industry, phosphorous based compounds (for example, phosphates, phosphonates etc.) used in detergent products can contribute to the above. Therefore, in pursuit of minimizing the environmental impact, the committee responsible for formulation of this standard has decided to revise it again. Hence, besides amalgamation of all amendments, the maximum limit of phosphate has been restricted in this revision. Further, the use of Zeolite, which is a non-phosphate based builder and use of co-builders enzymes (bio-based actives) for detergent has been recommended as an alternative to phosphate based builder. Tri-sodium citrate has also been incorporated in list of conventional builders and additives since it has the potential to replace phosphates and is more environment friendly than the phosphates.

It is necessary that the raw materials used in the formulation of detergents are such that in the concentration in which they will be present in the finished product, after interaction between them are free from any harmful effects. For determining the suitability of a new formulation or of a new raw material used in formulations for skin safety, necessary tests as prescribed in IS 11601 : 2002 Methods of safety evaluation of synthetic detergents — Tests for skin irritation and sensitization potential of synthetic detergents need to be followed.

A scheme for labelling environment friendly products to be known as ECO Mark has been introduced at the instance of Ministry of environment, Forest and Climate Change (MoEF&CC). The ECO Mark shall be administered by the Bureau of Indian Standards (BIS) under the *BIS Act*, 2016 as per the Resolution No 71 dated 20 February 1991 published in the Gazette of the Government of India. For a product to be eligible for ECO Mark, it shall carry the standard mark of BIS for quality besides meeting additional optional environment friendly (EF) requirements.

This standard covers **6.1** which calls for an agreement between the purchaser and the supplier.

This standard was formulated by CHD 25 technical committee. The list of experts who made significant contribution to the revision of this standard is given at Annex M.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

HOUSEHOLD LAUNDRY DETERGENT POWDERS — SPECIFICATION

(*Fifth Revision*)

1 SCOPE

This standard prescribes requirements, methods of sampling and tests for laundry detergent powders for household use.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the Indian Standards indicated below:

<i>IS No</i>	<i>Title</i>	<i>IS No</i>	<i>Title</i>
199:1989	Textiles — Estimation of moisture, total size or finish, ash and fatty matter in grey and finished cotton textile materials (<i>third revision</i>)	7597 : 2001	Glossary of terms relating to surface active agents (<i>first revision</i>)
264 : 2005	Nitric acid — Specification (<i>third revision</i>)	8401 : 1994	Alkyl benzene sulphononic acid (acid slurry) — Specification (<i>first revision</i>)
265 : 1993	Hydrochloric acid — Specification (<i>fourth revision</i>)	9458 : 1994	Synthetic detergents for washing woollen and silk fabrics — Specification (<i>first revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)	9985 : 1992	Sodium alkyl benzene sulphonate, technical — Specification (<i>first revision</i>)
4262 : 2002	Sulphuric acid — Code of safety (<i>first revision</i>)	11601 : 2002	Methods of safety evaluation of synthetic detergents — Tests for skin irritation and sensitization potential of synthetic detergents (<i>first revision</i>)
4707 (Part 1) : 2017	Classification for cosmetics raw materials and adjuncts: Part 1 Colourants (<i>third revision</i>)	12795 : 1989	Linear alkyl benzene — Specification
(Part 2) : 2017	Classification for cosmetic raw materials and adjuncts: List of raw materials generally not recognized as safe for use in cosmetics (<i>fourth revision</i>)	13933 : 1995	Method of test for ready biodegradability of surface active agents (modified Sturm test)
5785 (Part 4) : 1976	Methods of performance tests for surface active agents: Part 4 Relative detergency (<i>first revision</i>)	15267 : 2003	Zeolite detergent grade — Specification

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7597 shall apply.

4 GRADES

The material shall be of the following three grades:

- a) Grade 1,
- b) Grade 2, and
- c) Grade 3.

5 REQUIREMENTS

5.1 Description

The material shall be in the form of a free flowing powder, free from any unpleasant odour, and shall possess good lathering and cleaning properties. In addition to moisture, it may contain substances, such as colouring matter, preservatives, powder conditioners, opacifiers and optical brightening agents.

5.2 Active Ingredients

The active ingredients used in the formulation of synthetic detergent powders shall comprise one or more of the surface active agents, namely, linear alkyl benzene sulphonate, secondary alcohol sulphate, fatty alcohol sulphate, fatty alcohol ethoxylate, salts of sulphated fatty alcohol ethoxylate, sodium alpha sulpho fatty acid esters, alpha olefin sulphonate, soap, sugar esters and other non-ionic detergents.

5.2.1 If sodium alkyl benzene sulphonate is used as the active ingredient, it shall be manufactured from alkyl benzene sulphonic acid conforming to IS 8401 for which linear alkyl benzene conforming to IS 12795 shall be the starting material.

5.2.2 All other active ingredients shall conform to the relevant Indian Standards, as and when available.

5.3 Formulation

In addition to the active ingredients specified in **5.2** and **5.2.2**, the formulation may contain one or more of conventional builders or additives as given in Annex A.

5.3.1 In case non-ionic active detergent is used, the total active ingredient shall be determined by the method given in Annex B of IS 9458. If soap is present in the

detergent formulation, the result as determined above will include non-ionic detergent and soap.

5.3.2 The material shall not contain any plastic microbeads or other synthetic abrasive material.

5.3.3 The material may contain permitted colour as given in IS 4707 (Part 1). The material shall not contain any ingredient above the limit as given in IS 4707 (Part 2).

5.4 The material shall pass the test for skin irritant and sensitization potential when evaluated as per the method prescribed in IS 11601.

NOTE — This requirement has also been identified as specific requirement for ECO Mark.

5.5 The synthetic detergent powders shall not cause any damage to the fabrics during wash.

5.6 The material shall also comply with the requirements given in Table 1.

5.7 Additional Requirements for ECO Mark

5.7.1 General Requirements

5.7.1.1 The product shall conform to the requirements for quality safety and performance prescribed under **5.1** to **5.6** except that for phosphate content which shall be substituted with alternate environment friendly builder(s) to maintain similar detergency when tested according to IS 5785 (Part 4).

5.7.1.2 The manufacturers shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the provisions of the *Water (Prevention and Control of Pollution) Act, 1974* and *Air (Prevention and Control of Pollution)*

Table 1 Requirements for Household Laundry Detergent Powders

(Clauses 5.6, 7.2.1 7.2.2, 7.3.1 and 8.1)

Sl No.	Characteristics	Requirements for			Method of Test, (Ref to Annex)
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Active ingredient percent by mass, <i>Min</i>	19	16	10	B
ii)	Total phosphates expressed as P_2O_5 content percent by mass, <i>Max</i> (see Note 1 and 2)	2.5	2.5	2.5	D
iii)	Sodium tripolyphosphate (STPP) percent by mass, <i>Max</i> (see Note 2)	2.5	2.5	2.5	E
iv)	Active alkalinity (ml of 0.1 N HCl to titrate 50 ml of 1 percent product solution to phenolphthalein end point), <i>Max</i>	15	20	30	F
v)	Percent detergency, <i>Min</i>	65	55	45	G
vi)	Ash built up percent, <i>Max</i>	1	5	10	H

NOTES

1 Total phosphate content including phosphate based builder (as recommended in Annex A) shall not exceed 2.5 percent by mass for grade I, grade II and grade III.

2 Use of Zeolite (see IS 15267) as an alternative to phosphate based builder is recommended.

Act, 1981 along with the authorization, if required under *Environment (Protection) Act*, 1986 while applying for ECO Mark.

5.7.2 Specific Requirements

5.7.2.1 The material shall not contain any phosphate when tested as per the method prescribed in Annex D. Any other substitute used shall be environmental friendly but should be in sufficient quantity to ensure similar performance of the product as compared to that of phosphates.

5.7.2.2 The material shall pass the test for skin irritant and sensitization potential when evaluated as per the method prescribed in IS 11601.

5.7.2.3 The surfactants used in the manufacture of household laundry detergent powders shall be readily biodegradable when tested by modified sturm test as prescribed in IS 13933.

6 PACKING AND MARKING

6.1 Packing

6.1.1 The material shall be suitably packed as agreed to between the interested parties. The packing shall be capable to prevent any moisture absorption.

6.1.2 For ECO Mark the product shall be packed in such packages which are made from recyclable/reusable or biodegradable materials and declared by the manufacturer and may be accompanied with detailed instructions for proper use of product.

6.2 Marking

6.2.1 Each package shall be securely closed and marked with the following information:

- a) Name and grade of the material;
- b) Indication of the source of manufacture;
- c) Net mass of the material, when packed;
- d) Batch number or lot number in code or otherwise;
- e) Month and year of manufacture;
- f) A cautionary notice: Detergent solutions can be skin irritants. Avoid prolonged contact. Rinse garments and hands thoroughly.
- g) The following critical ingredients in descending order of quantity, percent by mass:
 - 1) Active ingredients,
 - 2) Builders,
 - 3) Soda ash,
 - 4) Fillers, and
 - 5) Enzymes, if any.

6.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6.2.3 The following information shall be marked on the label for ECO Mark:

- a) List of identified critical ingredients in descending order of quantity, percent by mass [see 6.2.1(g)]; and
- b) The criteria for which the product has been labelled as ECO Mark.

NOTE — Loss in mass, even in packed condition, may occur on account of moisture loss due to environmental conditions.

7 SAMPLING

7.1 General

General precautions for drawing samples, its scale and preparation of test samples shall be as prescribed in Annex J.

7.2 Number of Tests

7.2.1 Tests for the estimation of characteristics prescribed at SI No. (i), (iii), (v) and (vi) of Table 1 shall be conducted on each of the individual samples separately.

7.2.2 Tests for the estimation of the remaining characteristics prescribed in Table 1 shall be conducted on the composite sample.

7.3 Criteria for Conformity

7.3.1 For Individual Samples

For each of the characteristics which has been determined on the individual samples (see 7.2.1) the mean (\bar{x}) and the range (R) of the test results shall be calculated as follows:

$$\text{Mean } (\bar{x}) = \frac{\text{Sum of test results}}{\text{Number of test results}}, \text{ and}$$

$$\text{Range (R)} = \text{Difference between the maximum and minimum value of the test results.}$$

The lot shall be deemed as conforming to the requirement if the expression ($\bar{x} - 0.4 R$) is greater than or equal to minimum value given at [SI No. (i) and (v)] of Table 1 and the expression ($\bar{x} + 0.4 R$) is less than or equal to maximum value given at [SI No. (vi)] of Table 1.

7.3.2 For Composite Sample

For declaring the conformity of the lot to the requirements of the other characteristics determined on the composite sample, the test results for each characteristic shall satisfy the relevant requirement.

8 TESTS

8.1 Tests to evaluate the characteristic prescribed in Table 1 shall be conducted as prescribed in Annexes B

to J. Reference to relevant clauses of Annex is given in col 6 of Table 1.

8.2 Quality of Reagents

Unless otherwise specified pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the results, of analysis.

ANNEX A

(Clause 5.3 and note under Table 1)

LIST OF CONVENTIONAL BUILDERS AND ADDITIVES

1 Crystalline sodium aluminosilicate (zeolite)	11 Optical brighteners
2 Trisodium phosphate	12 Lather boosters
3 Sodium carbonate	13 Sodium sulphate/sodium chloride
4 Tetra sodium pyrophosphate	14 Perfume
5 Sodium tripolyphosphate	15 Preservatives
6 Sodium hexametaphosphate	16 Chelating agents (sequestering agents)
7 Sodium carboxymethyl cellulose	17 Colours
8 Sodium silicate	18 Enzymes
9 Sodium bicarbonate	19 Bleach and bleach activators
10 Borax	20 Tri sodium citrate

ANNEX B

[Clause 8.1, and Table 1, Sl No. (i)]

DETERMINATION OF ACTIVE INGREDIENT BY CATION TITRATION**B-1 GENERAL**

In this prescribed method, the molecular mass of active matter has been taken as 342. In practice, the molecular mass of sodium alkyl benzene sulphonate varies from 337 to 347 depending on the molecular mass of alkyl benzene used for sulphonation. This method shall therefore, be used for routine analysis. In case of any dispute or doubt, the molecular mass of sodium alkyl benzene sulphonate shall be determined by the method prescribed in Annex C and which then used in calculating the active ingredient content by this method.

B-2 OUTLINE OF THE METHOD

A solution of the anionic detergent is shaken with methylene blue in chloroform, which dissolves the methylene blue salt of the detergent. The mixture is titrated with a cationic active agent which combines with all the free anions of detergent and begins to displace methylene blue from the salt. The end point is taken when sufficient methylene blue has been displaced into the aqueous layer to produce phases of equal colour intensity. As the reaction is not stoichiometric, it is essential to carry out standardization using a known anionic detergent similar in nature to the unknown.

NOTE — Hypochlorites and sulphites interfere with detection of the end point and should be destroyed by the addition of ferrous sulphate and hydrogen peroxide respectively.

B-3 APPARATUS

B-3.1 Volumetric Flasks, 1 000 ml, 500 ml and 250 ml capacity

B-3.2 Stoppered Graduated Cylinder, 100 ml capacity

B-3.3 Graduated Cylinder, 50 ml capacity

B-3.4 Burette, 25 ml capacity

B-3.5 Pipette, 10 ml capacity

B-3.6 Beaker, 250 ml capacity

B-4 REAGENTS

B-4.1 Chloroform, chemically pure

B-4.2 Sulphuric Acid, 5 N

Carefully and slowly add 134 ml of sulphuric acid (relative density 1.84) to 300 ml of water with continuous stirring and cool to room temperature and dilute to 1 litre.

CAUTION — Highly corrosive, causes severe burns. Use necessary safety implements (see IS 4262).

B-4.3 Standard Sulphuric Acid, 1.0 N

B-4.4 Standard Sodium Hydroxide Solution, 1.0 N

B-4.5 Standard Sodium Lauryl Sulphate Solution, 0.004 M

Check up purity of sodium lauryl sulphate as given in **B-4.5.1** and simultaneously prepare the standard solution.

B-4.5.1 Determination of Purity of Sodium Lauryl Sulphate

Weigh 5 ± 0.2 g of sodium lauryl sulphate to the nearest 1 mg into a 250 ml round bottom flask with ground glass neck. Add exactly 25 ml of standard sulphuric acid (see **B-4.3**) and reflux under water condenser. During the first 5 to 10 min, the solution will thicken and tend to foam strongly which is controlled by removing the source of heat and swirling the contents of the flask in order to avoid excessive foaming, instead of refluxing. The solution may be left on a boiling water bath for further 10 min. After the foaming ceases and solution clarifies, reflux the solution for further one and half hours. Remove the source of heat, cool the flask and carefully rinse the condenser with 30 ml of ethanol followed by water. Add a few drops of phenolphthalein indicator solution and titrate with standard sodium hydroxide solution. Carry out a blank test by titrating 25 ml of standard sulphuric acid (see **B-4.3**) with standard sodium hydroxide solution.

$$\text{Sodium lauryl sulphate content, percent by mass} = \frac{28.84(V_1 - V_0)N_1}{m_1}$$

where,

V_0 = volume of standard sodium hydroxide solution used for the blank, in ml;

V_1 = volume of standard sodium hydroxide solution used for the sample, in ml;

N_1 = normality of standard sodium hydroxide solution; and

m_1 = mass of sodium lauryl sulphate taken for the test, in g.

B-4.5.2 Procedure

Weigh 1.14 to 1.16 g of sodium lauryl sulphate to the nearest 1 mg and dissolve in 200 ml of water. Transfer to a ground-glass stoppered one litre volumetric flask and dilute to the mark with water. Calculate the molarity, M_1 of the solution as follows:

$$M_1 = \frac{m_2 \times \text{purity, percentage by mass}}{288.4 \times 100}$$

where,

m_2 = mass of sodium lauryl sulphate taken, in g.

B-4.6 Standard Benzethonium Chloride Solution — 0.004 M

Weigh 1.75 to 1.85 g of benzethonium chloride to the nearest 1 mg and dissolve in water. Transfer to a ground

glass-stoppered 1 litre volumetric flask and dilute to the mark with water.

NOTES

1 In order to prepare a 0.004 M solution, dry the benzethonium chloride at 105°C. Weigh 1.92 g to the nearest 1 mg, dissolve in water and dilute to 1 litre. While drying care shall be taken that temperature should not exceed beyond 105°C.

2 Other cationic reagents, such as cetyltrimethyl ammonium bromide give results identical to those obtained using benzethonium chloride. However, these tests have not been carried out in sufficient number to make it possible to state that the results will be identical no matter what the product analysed for that reason. If benzethonium chloride is not available, it is permitted to use another reagent and same should be stated in the test report. However in case of any dispute, only benzethonium chloride shall be used.

B-4.7 Phenolphthalein Indicator Solution

Dissolve 1 g of phenolphthalein in 100 ml of 95 percent (v/v) ethanol.

B-4.8 Methylene Blue Solution (0.005 Percent)

Dissolve 0.05 g of methylene blue, 50 g of sodium sulphate and 6.8 ml of concentrated sulphuric acid in water and make up the volume to 1 l with water.

B-4.9 Sample Solution

Weigh a suitable quantity of the sample measured quantity of water so that resultant solution contains 100 to 160 mg of anionic active matter per 100 ml. About 0.65-0.70 g of sodium alkyl benzene sulphonate (based on 100 percent purity) or 4.5 g of the sample having around 16 percent anionic active matter content per 500 ml of the solution is suitable.

B-5 PROCEDURE**B-5.1 Standardization of Benzethonium Chloride Solution**

B-5.1.1 Pipette 10 ml of standard sodium lauryl sulphate solution (see **B-4.5** and **B-4.5.2**) in a 100 ml graduated cylinder provided with a glass stopper. Add 15 ml of chloroform and 25 ml of methylene blue solution to the cylinder. Shake well. The chloroform layer (lower) will be coloured blue or greenish blue.

B-5.1.2 From the burette, add benzethonium chloride solution slowly, initially in portions of 0.2 ml at a time. After each addition, stopper the cylinder, shake well and allow the phases to separate. Initially the chloroform phase will be coloured blue or greenish blue. Towards the end, the colour would start migrating to the aqueous layer. Note the reading at which the colour intensity in both the phases is the same when viewed under standard conditions of light, for example, against a white porcelain tile, under normal daylight.

B-5.1.3 Calculate the molarity of benzethonium chloride solution as follows:

Molarity of benzethonium chloride solution,

$$M_2 = \frac{10M_1}{V_1}$$

where

M_1 = molarity of sodium lauryl sulphate solution (B-4.5.2), and

V_1 = volume of benzethonium chloride solution added, in ml.

B-5.2 Determination of Anionic Active Matter

Take 10 ml of the sample solution (B-4.9) instead of sodium lauryl sulphate solution and proceed as described in B-5.1.

B-5.3 Calculate the anionic active matter as sodium alkyl benzene sulphonate as follows:

Anionic active matter, percent by mass =

$$\frac{342 \times V_2 \times M_2 \times 5}{m_2}$$

where

342 = molecular mass of sodium alkyl benzene sulphonate taken for calculation,

V_2 = volume of benzethonium chloride solution added, in ml,

M_2 = molarity of benzethonium chloride solution (B-5.1.3), and

m_2 = mass of the sample, in g.

ANNEX C

(Clauses 8.1 and B-1)

DETERMINATION OF MOLECULAR MASS OF SODIUM SALT OF ALKYL BENZENE SULPHONIC ACID

C-1 APPARATUS

C-1.1 Beakers, 150 and 1 000 ml capacity.

C-1.2 Buchner Flask, 500 ml capacity, fitted with a sintered glass filter funnel (porosity 4).

C-1.3 Separating Funnels, 1 000 ml capacity.

C-1. 4 Wide-Mouthed Flat-Bottom Flask, 200 ml capacity.

C-1.5 Air-Oven, preferably electrically heated with temperature control and display device.

C- 1.6 Steam Bath

C-2 REAGENTS

C-2.1 Caustic Soda Solution, 10 percent (m/v)

C-2.2 Ethyl Alcohol, 30 percent, 96 percent and absolute (v/v).

C-2. 3 Diethyl Ether

C-2.4 Acetone

C-2.5Phenolphthalein Indicator, 1 percent solution in 95 percent (m/v) ethyl alcohol.

C-2.6 Methyl Orange Indicator, 0.1 percent (m/v).

C-2.7 Ferric Ammonium Sulphate Indicator, saturated solution.

C-2.8 Standard Sulphuric Acid, approximately 0.1 N.

C-2.9 Standard Silver Nitrate Solution, approximately 0.1 N.

C-2.10 Standard Ammonium Thiocyanate Solution, Approximately 0.1 N.

C-2.11 Nitric Acid, Concentrated, relative density 1.42.

C-2.12 Nitrobenzene

C-3 PROCEDURE

C-3.1 Weigh about 3 g of the material into a 150 ml beaker. Dissolve in minimum quantity of water and neutralize with caustic soda solution, if required. Evaporate on a steam bath to almost complete dryness. Digest with 50 ml of 96 percent ethyl alcohol by heating on a steam bath for about 2 min. Stir and break up any hard lumps with a glass rod flattened at one end. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Repeat the alcoholic digestion in a similar manner with 5 further consecutive 30 ml portions of boiling ethyl alcohol. Filter each extract in turn through the same sintered glass funnel and finally, wash the residue five times with hot ethyl alcohol to remove all the alcohol soluble. Evaporate the combined filtrate to approximate 50 ml in an evaporating dish and transfer it to a separating funnel. Rinse the evaporating dish once with 50 ml of 96 percent ethyl alcohol and then four times with 50 ml portions of water. Add each wash in turn to the separating funnel. Add 150 ml of diethyl ether,

swirl gently to ensure adequate mixing, and allow the two phases to separate. Run off the aqueous alcoholic layer into a second separating funnel, and extract twice with 75 ml portions of diethyl ether. Transfer the aqueous alcoholic phase into a beaker, and combine the three ether extracts.

C-3.2 Take the combined ether extracts in a clean separating funnel. Wash three times successively with 50 ml portions of 30 percent ethyl alcohol and then successively with 50 ml portions of water until the ether phase is free from alcohol, usually 7 to 10 water washes are necessary. Combine all the alcoholic and aqueous extracts, neutralize to phenolphthalein and evaporate on a steam bath until the volume is reduced to about 25 ml. Add an equal volume of absolute alcohol and evaporate to dryness. The solution should remain pink to phenolphthalein throughout evaporation. To ensure that the residue is completely anhydrous, add 30 ml of hot absolute alcohol and again evaporate to dryness. Extract the residue with 30 ml of hot 96 percent ethyl alcohol, stirring and breaking up the solid matter in the dish with a glass rod. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Extract the residue in the dish with further six consecutive 30 ml portions of hot 96 percent alcohol. Pass each extract in turn through the sintered glass filter. Finally, wash the residue in the sintered glass filter three times with about 20 ml of hot 96 percent ethyl alcohol from the jet of a wash bottle.

C-3.3 Transfer the filtrate and washing in the Buchner flask to a wide-mouthed flat-bottom flask, evaporate nearly to dryness on a water-bath, and drive off the remaining solvent by directing a gentle stream of dry air into the flask whilst continuously rotating the latter on the water bath, a thin film of active matter, easy to dry, is thereby obtained. Add 10 ml of acetone, evaporate and remove the last traces of solvent as prescribed above, cool in a desiccator and weigh. Heat the flask for not more than 5 min in an air oven at a temperature of $100 \pm 1^\circ\text{C}$, gently blow out with a current of air, cool and re-weigh. Repeat this drying process until the difference between two successive weighing does not exceed 3 mg.

C-3.4 The extract obtained contains active matter, some sodium chloride and possible traces of alkali carbonates which may have passed through the filter in the presence of the detergent. Find the percentage of sodium carbonate and sodium chloride in the extract by using a portion of the extract as prescribed in **C-3.4.1** and **C-3.4.2** respectively.

C-3.4.1 Determination of Alkali Carbonates

Weigh accurately about 1 g of the extract. Dissolve it in cold water, add a few drops of methyl orange indicator and titrate with standard sulphuric acid to methyl orange end point.

C-3.4.1.1 Calculation

Mass of the sodium carbonate, in g = $\frac{0.053 \times V_1 \times N_1 \times m_1}{m_2}$

where

V_1 = volume of standard sulphuric acid solution used, in ml;

N_1 = normality of the standard sulphuric acid solution;

m_1 = mass in g, of the total extract; and

m_2 = mass in g, of the extract taken for analysis.

C-3.4.1.2 Preserve the solution for the estimation of chlorides.

C-3.4.2 Determination of Chlorides

To the solution remaining after the estimation of alkali carbonate (*see C-3.4.1.2*) add 2 ml of concentrated nitric acid and 20 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene and shake vigorously. Titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate as indicator.

C-3.4.2.1 Calculation

Mass in g of sodium chloride =

$$\frac{0.0585 \times (20N_3 - V_2N_2) \times m_1}{m_2}$$

where

N_3 = normality of standard silver nitrate solution;

V_2 = volume of standard ammonium thiocyanate solution used, in ml;

N_2 = normality of standard ammonium thiocyanate solution;

m_1 = mass of the total extract (*see C-3.3*), in g; and

m_2 = mass of the extract taken for analysis in **C-3.4.1.1**, in g.

C-3.5 Weigh accurately about 0.65-0.70 g of the extract (*see C-3.4*). Dissolve in water and make up to 500 ml. Follow the titration procedure given in **B-5.1** taking 10 ml of the solution for titration.

C-4 CALCULATION

Molecular mass of sodium salt of sulphonic acid =

$$\frac{100 \times m}{5 \times V_1 \times M_1}$$

where

m = mass of the extract taken after correcting for sodium carbonate and sodium chloride, in g;

V_1 = volume of benzethonium chloride solution added, in ml and

M_1 = molarity of benzethonium chloride solution.

ANNEX D

[Clause 8.1, and Table 1, Sl No. (ii)]

DETERMINATION OF TOTAL PHOSPHATES

D-1 GENERAL

The sample is oxidized by gently heating with sodium nitrate. Silica is removed and the condensed phosphates are hydrolysed and precipitates are heteropoly acid and not their salt. Heteropoly acid react with alkali to form individual salt of each constituent acid of heteropoly acids and precipitated as phosphomolybdate by addition of ammonium molybdate in acidic condition. Precipitate is washed with dilute potassium nitrate solution and phosphorus is determined by titration of precipitated phosphomolybdate with standard sodium hydroxide using phenolphthalein as indicator.

D-2 APPARATUS

D-2.1 Silica Dish, 7 cm diameter

D-2.2 Beaker, 250 ml capacity

D-2.3 Buchner Flask, 500 ml capacity with a sintered glass filter funnel

D-2.4 Volumetric Flask, 500 ml capacity.

D-2.5 Funnel, 7.5 cm diameter.

D-2.6 Wide-Mouthed Flat Bottom Flask, 500 ml capacity.

D-3 REAGENTS

D-3.1 Sodium Nitrate

D-3.2 Hydrochloric Acid concentrated (see IS 265)

D-3.2.1 Dilute Hydrochloric Acid

D-3.3 Nitric Acid (see IS 264)

D-3.4 Ammonium Molybdate Reagent

Dissolve 90 g of ammonium molybdate in hot water. Add 240 g of ammonium nitrate and stir to dissolve. Cool and add 30 ml of concentrated ammonia (0.9 relative density). Dilute to 1 litre.

D-3.5 Potassium Nitrate, 1.25 percent solution in water

D-3.6 Standard Sodium Hydroxide Solution, 1 N

D-3.7 Standard Sulphuric Acid, 1 N

D-3.8 Phenolphthalein Solution, 1 percent solution (m/v) in ethyl alcohol.

D-3.9 Potassium Hydroxide Solution, 0.1 N

D- 3.10 Sodium Carbonate

D-4 PROCEDURE

D-4.1 Weigh accurately about 1.5 g of the sample in a silica dish and mixed with about twice sample weight of equal parts of sodium carbonate and sodium nitrate mixture and heat gently over a bunsen burner until the sample is completely oxidized. Cool the mixture and add 15 ml of concentrated hydrochloric acid to dissolve the mass and evaporate to dryness. Add further 15 ml of concentrated hydrochloric acid and repeat the evaporation procedure. Finally extract the residue in 25 ml of 1:1 hydrochloric acid solution and filter the residue on filter paper through Whatman 40 filter paper. Wash 4 times with 50 ml of water. Collect the filtrate and washings and make up to 250 ml in a volumetric flask.

D-4.2 Pipette out 50 ml aliquot of solution in a 250 ml beaker. Add 10 ml of nitric acid and boil for 15 min. Cool and add 100 ml of water and adjust the temperature of the solution to 40 to 45°C. Add 50 ml of ammonium molybdate solution (previously heated to 40°C) slowly with constant stirring. Allow to stand for 30 min. Filter the precipitate through a quantitative filter paper and wash with 1.25 percent potassium nitrate solution till 5 ml of the filtrate with one drop of phenolphthalein does not require more than 3 to 4 drops of 0.1 normal caustic potash solution to produce pink colour. Transfer the filter along with precipitate to a 500 ml wide mouth flat-bottom flask and add 100 ml water. Heat over a water-bath for 15 min, cool and titrate with 1 N sodium hydroxide solution using 1 ml of phenolphthalein till the pink colour just appears, add 2 ml excess of sodium hydroxide solution. Shake well, heat to 60°C in a water-bath. Cool the solution and back titrate against 1 N sulphuric acid till the pink colour just disappears. Find the volume of normal sodium hydroxide solution required to react with the precipitate.

D-5 CALCULATION

Total phosphates as P_2O_5 , percent by mass =

$$\frac{V \times N \times 0.001349 \times 250 \times 100 \times 140}{m \times 5062}$$

where

V = volume of sodium hydroxide solution required to react with the precipitate, in ml;

N = normality of sodium hydroxide solution, and

m = mass of sample taken for test in g.

ANNEX E

[Clause 8.1, and Table 1, Sl No (iii)]

DETERMINATION OF SODIUM TRIPOLYPHOSPHATE (STPP) CONTENT

E-1 GENERAL

The procedure based on ion-exchange and paper chromatography for determination of sodium tripolyphosphate (STPP) are time consuming and lengthy. The gravimetric method prescribed here is simple, quick and does not require any special facilities.

E-2 OUTLINE OF THE METHOD

It is based on the quantitative precipitation of STPP by tris (ethylenediamine) cobalt (III) chloride as $\text{Co(en)}_3\text{-H}_2\text{P}_3\text{O}_{10}\cdot 2\text{H}_2\text{O}$ at 3.5 pH after removing the active detergent, by dissolution in alcohol, and water insoluble silica/silicates.

E-3 REAGENTS

E-3.1 *p*-Nitrophenol Indicator Solution, dissolve 0.1 g of *p*-nitrophenol in 100 ml of water.

E-3.2 Dilute Hydrochloric Acid, 0.5 N approximately.

E-3.3 Acetate Buffer Solution pH 3.5, dissolve 52.6 ml of glacial acetic acid and 6.16 g anhydrous sodium acetate in water and dilute to 500 ml.

E-3.4 Sodium Tripolyphosphate Hexahydrate ($\text{Na}_3\text{P}_3\text{O}_{10}\cdot 6\text{H}_2\text{O}$), crystallize from commercially available STPP as STPP Hexahydrate as per method prescribed in E-3.4.1 and E-3.4.2.

E-3.4.1 Procedure

Weigh 150 g of commercial sodium tripolyphosphate, using material of low metaphosphate content. Dissolve in 1 200 ml of water, and filter to remove any suspended material. Add 300 ml of denatured spirit slowly, continuously stir, over a period of 2 h. Separate the crystals of hexahydrate by filtration using a Buchner funnel fitted with a glass disc of medium porosity. Wash the crystals successively with 100 ml each of 25 percent, 50 percent, 75 percent and 90 percent (v/v) solutions of denatured spirit in water.

E-3.4.2 Dissolve the crystals of hexahydrate (see E-3.4.1) in 900 ml of water and salt out with 225 ml of denatured spirit using the same procedure as described above and again perform filtering and washing the crystals with the same volumes of dilute denatured spirit. Repeat the recrystallization using 675 ml of water and 170 ml of denatured spirit. Recrystallize once more using 500 ml of water and 150 ml of denatured spirit. After filtering and washing, air dry the crystals of hexahydrate for several days.

E-3.5 Tris (Ethylenediamine) Cobalt (III) Chloride

Prepare as per method explained in E-3.5.2.1 to E-3.5.2.4. Use 15 to 20 percent solution of the reagent.

E-3.5.1 Reagents

- Diaminoethane hydrate (ethylene diamine);
- Concentrated hydrochloric acid (see IS 265);
- Cobalt chloride ($\text{CoCl}_2\cdot 6\text{H}_2\text{O}$); and
- Denatured spirit.

E-3.5.2 Procedure

E-3.5.2.1 Weigh 261 g of diaminoethane hydrate into a 1 000 ml beaker. Partly neutralize with 85 ml of concentrated hydrochloric acid in 535 ml of water. Pour the mixture into a solution of 250 g of cobalt chloride in 750 ml of water in a conical flask with constant agitation/stirring. Pass a vigorous stream of air into the solution through a gas distribution tube for 8 h.

E-3.5.2.2 Transfer the solution to a 2 000 ml beaker and evaporate on a steam-bath under a stream of air until crystals form on the surface of the solution. Cool the solution and add 150 ml of concentrated hydrochloric acid under constant stirring. Warm until the crystals re-dissolve. Salt out the trisdiaminoethane cobalt(III) chloride by slowly adding of 300 ml of denatured spirit over a period of 1 h with constant stirring of solution. Cool and filter through a Buchner funnel and suction. Discontinue suction and add 150 ml of denatured spirit to the funnel and stir to wash the crystals. Re-apply suction and filter. Repeat the washings three more times. Suck dry, spread out the crystals in an evaporating dish and allow the denatured spirit to evaporate.

E-3.5.2.3 Dissolve the crystals obtained in E-3.5.2.2 above in 200 ml of boiling water in 1 000 ml beaker with constant stirring. If the crystals do not dissolve in this volume of water, add 20 ml portion of water, reheating to boiling after each addition, until complete solution is obtained. Remove from the source of heat and salt out with 300 ml of the denatured spirit as explained above. Filter and wash as before. If four washings do not give a colourless filtrate, continue washing until the filtrate is colourless.

E-3.5.2.4 Spread out the crystals in a thin layer in large evaporating dishes and allow to air-dry overnight and then dry in an oven at 100°C overnight.

NOTE — These crystals are hygroscopic and should be freshly dried each time before weighed to prepare fresh solution. An yield of about 300 g of the dried crystals is obtainable.

E-4 PROCEDURE

E-4.1 Determination of STPP in Recrystallized Sodium Tripolyphosphate Hexahydrate ($\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$)

To the solution containing 0.2 to 0.4 g of STPP (accurately weighed) in 75 ml water, add 2 to 3 drops of *p*-nitrophenol indicator. Add dilute hydrochloric acid dropwise with constant stirring till the indicator changes to colourless. Add 10 ml of the buffer solution to get the desired *pH* of about 3.5. Add 20 ml of isopropanol. Warm the solution at 40°C in a water bath for 15 min. Add 10 ml of cobalt reagent in portions ; first 4 ml, stir vigorously for 1 min, another 4 ml, stir again for 1 min and finally add the remaining 2 ml and stir for 2 min. Allow the precipitate to stand at 40°C for 15 min. Filter through a dry and weighed sintered glass crucible. Wash with water till the washings are free of chloride ions. Dry the crucible in an oven at 110°C for 45 min. Cool in a desiccator and weigh till constant mass is obtained.

E-4.1.1 Calculation

Purity of recrystallized STPP, percent by mass =

$$\frac{M_2 \times 0.6495 \times 100}{M_1}$$

Where

M_2 = mass of the cobalt tripolyphosphate precipitate obtained, in g, and

M_1 = mass of recrystallized sample of STPP taken in the test, in g.

E-4.2 Determination of Tripolyphosphate Content in Detergent Powder

E-4.2.1 Separation of Active Detergent

Treat an accurately weighed quantity (about 3 g) of detergent powder with 10 ml of water in a 500 ml beaker. Add 75 ml of denatured spirit and stir for 10 min. Filter through a fluted filter paper (Whatman No 41 or equivalent) by decantation. Treat the residue left in the beaker with three more portions of 75 ml denatured spirit, followed by stirring and filtration by decantation after each addition. Discard the filtrate.

E-4.2.2 Preparation of STPP Sample Solution

Wash down the residue on the filter paper into the beaker containing most of the inorganic material using

a jet from a wash bottle. Also wash down the material sticking to the sides of the beaker with water, using in all about 75 ml of water. Stir and keep aside for 15 min with occasional stirring. Filter through filter paper (Whatman No. 4 or equivalent) transferring all solids into the filter paper with help of minimum water. Wash the residue with 25 ml of water and collect the washings and the filtrate in a 500 ml beaker for STPP determination. Add a known amount of the recrystallized STPP (0.150 to 0.25 g), (*see* Note) and stir to dissolve. Add 3 to 4 drops of *p*-nitrophenol indicator. Green colour will appear. Add dilute hydrochloric acid drop wise with stirring till the indicator colour disappears. Add 10 ml of acetic acid buffer to get 3.5 *pH*. Add 15 ml of isopropanol and place the beaker in a warm waterbath (40°C) for 15 min.

NOTE — In place of adding STPP, preweighed crystals of tris (ethylenediamine) cobalt tripolyphosphate obtained from previous analysis if any, may be added before adding the cobalt reagent and finally subtracted from the mass of the precipitate for the purpose of calculation.

E-4.2.3 Precipitation of STPP by the Cobalt Reagent

Add 10 ml of cobalt reagent solution in portions in three instalments, first add 4 ml and stir vigorously for 2 min, then add 4 ml and again stir vigorously for 2 min and finally add 2 ml and stir for 1 min. Place the beaker containing the precipitate on water-bath (40°C) for 10 min. Add 0.15 to 0.25 g of filter aid and stir. Filter through a weighed sintered glass crucible. Wash the precipitate with water till washings are free of chloride ions. Dry the precipitate at 110°C for 1 h. Weigh till constant mass (M_4) is obtained.

E-5 CALCULATION

E-5.1 STPP in detergent powder, percent by mass =

$$\frac{[(M_4 - M_3) \times 0.6945] - M_2}{M_1} \times 100$$

where

M_4 = mass of the filter-aid and precipitate obtained, in g;

M_3 = mass of filter-aid added, in g;

M_2 = mass of STPP or $[\text{Co(en)}]\text{Cl}_3$ added (calculated as anhydrous STPP), in g; and

M_1 = mass of sample of powder taken in E-4.2.1, in g.

ANNEX F

[Clause 8.1, and Table 1, Sl No (iv)]

DETERMINATION OF ACTIVE (RESERVE) ALKALINITY**F-1 APPARATUS****F-1.1 pH Meter****F-1.2 Beaker**, 100 ml capacity**F-1.3 Magnetic Stirrer****F-1.4 Burette****F-2 REAGENTS****F-2.1 Hydrochloric Acid**, 0.1 N**F-3 PROCEDURE**

Weigh 10.0 g sample in 500 ml water and make up the

volume to 1000 ml with water. Shake well and pipette out 50 ml of solution in a beaker. Place the beaker on amagnetic stirrer and mix the contents thoroughly. Note down the pH of the solution using a pH meter. With the electrode of the pH meter dipping in the solution and keeping the pH meter 'ON', add drop by drop 0.1 N hydrochloric acid from a burette till the pH of the solution drops to 8. While adding hydrochloric acid stir the solution continuously. Note the amount of 0.1 N hydrochloric acid required to bring down the pH of the solution to 8 which is a measure of the active alkalinity of the test sample.

Mean of 2 replicate measurements will give active (reserve) alkalinity expressed as amount in ml of 0.1 N hydrochloric acid.

ANNEX G

[Clause 8.1, and Table 1, Sl No (v)]

PROCEDURE FOR DETERGENCY TEST (POWDERS)**G-1 OUTLINE OF THE METHOD**

The method prescribed here is based on the use of Terg-O-tometer. Cloth is artificially soiled and the soil is removed by washing the soiled swatches of cloth with a solution of the detergent powder to be evaluated under standard conditions. A control detergent powder (see below for details) is also run simultaneously. The reflectance of the unsoiled, soiled and washed swatches is measured instrumentally using a standard photoelectric reflection meter. The detergency is expressed as percentage of soil removal.

G-2 APPARATUS**G-2.1 Photoelectric Reflection Meter**

With built-in galvanometer and tungsten lamp as an illuminant.

G-2.2 Terg-O-tometer

With a battery of four or six agitator washers in 2 l stainless steel beakers. The angle through which the agitators oscillated is 350°. The speed of rotation is adjustable and set at 100 strokes per minute, each back and forth movement representing one stroke. The beakers are fully immersed in an electrically controlled water-bath. The agitators and beakers are removable.

G-2.3 Cloth Soiling Machine

Electrically operated mangle with variable pressure arrangements which can be recorded and variable speed drive with attached air-drying chamber fitted with exhaust.

G-3 PREPARATION OF STANDARD SOILED CLOTH TEST SPECIMENS

Prepare soiled cloth swatches as per 6 of IS 5785 (Part 4).

G-4 CONTROL POWDER FORMULATION

AD	19
STPP	05
Zeolite	10
Co-builder-Enzyme	0.4
Soda	10
Alk silicate	10
SCMC	1
Na ₂ SO ₄	40
Moisture	4.6
	= 100

G-5 WASHING PROCEDURE

G-5.1 The ratio of cloth (in g) to the volume of solution (in ml) shall be 1:100.

G-5.2 Prepare 1 l solution of 0.5 percent in 300 ppm water considering the basis of product concentration used in washing. Introduce 5 soiled swatches into beakers and agitate. Wash the specimens for exactly 10 min at $27 \pm 2^\circ\text{C}$.

G-5.3 Remove the beaker from the water-bath and decant the solution and rinse 3 times with 300 ppm water.

G-5.4 Repeat with 5 more wash loads of 5 swatches each, for each formulation and control powder. Total of six replicate washings is given so that at the end, all the formulations are washed in all beakers.

G-5.5 Air dry washed swatches and measure their reflectance.

G-6 EVALUATION OF SOIL REMOVAL

Fold the desired unsoiled cloth, soiled swatches and washed swatches into four folds so as to minimize the effect of colour of background and take reflectance measurements. Operate the reflectometer in accordance with the instruction supplied with the instrument.

G-7 CALCULATION AND REPORTING

The detergency value expressed as percentage soil removed is calculated from the following equation.

$$\text{Percentage of soil removal} = \frac{W_o}{S_o} \times 100$$

where,

$$W_o = \frac{(100 - R_w)^2}{200R_w} - \frac{(100 - R_3)^2}{200R_3}$$

$$S_o = \frac{(100 - R_c)^2}{200R_c} - \frac{(100 - R_3)^2}{200R_3}, \text{ and}$$

R_c , R_w and R_3 are reflectances of clean, washed and soiled fabric pieces respectively.

Analysis of variance to be used for the handling of data and obtaining significant differences.

G-8 TREATMENT OF DATA AND REPORTING

Assign a value of 70 percent detergency to control powder. Normalise the detergency values of products by applying a correction factor, that is, 70 percent detergency value for control powders and report the normalised detergency value.

ANNEX H

[Clause 8.1, and Table 1, Sl No (vi)]

DETERMINATION OF ASH BUILT UP ON FABRICS

H-1 GENERAL

This test determines the built-up of ash on a fabric under the condition of test.

H-2 REAGENTS

H-2.1 Sodium Hexametaphosphate

H-2.2 Hard Water, of 300 ppm (as CaCO_3), made by adding 2.96 g of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ and 2.64 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ to 10 l of water.

H-2.3 Bleached Cotton Long Cloth ($125 \pm 25 \text{ g/m}^2$)

H-3 EQUIPMENT

H-3.1 Washing Appliance, like standard Terg-O-tometer.

H-3.2 Air Oven, with arrangements for drying of washed swatches by hanging.

H-4 PROCEDURE

H-4.1 Cut the cotton cloth into swatches of $10 \text{ cm} \times 10 \text{ cm}$ size. 10 swatches are required for each set.

H-4.2 Number the raw swatches and wash them 2 times in a Terg-O-tometer with sodium hexametaphosphate solution (3 g/l) to remove the original salts present in the swatches. Rinse the swatches thoroughly with distilled water. Dry the swatches in an air oven at 105°C .

H-4.3 Stir 10 prewashed swatches (*see H-4.2*) in 1 litre solution of 0.2 percent concentration of the test detergent product prepared in 300 ppm hard water at $27 \pm 2^\circ\text{C}$ for 20 min in a Terg-O-tometer. Rinse the swatches in 1 litre hard water of hardness 300 ppm by stirring in the Terg-O-tometer for 10 min at $27 \pm 2^\circ\text{C}$.

H-4.4 Dry the swatches at 50°C in an air oven. Repeat the washing, rinsing and drying operations 25 times. After 25 washes, dry the swatches in an air oven at 105°C . Determine the ash content of both prewashed (*see H-4.2*) and after washed fabric with detergent sample as explained above at 800°C as per IS 199.

ANNEX J

(Clauses 7.1 and 8.1)

SAMPLING PROCEDURE FOR SYNTHETIC DETERGENTS

J-1 GENERAL REQUIREMENTS

J-1.1 Following precautions shall be taken in drawing, preparing, storing and handling samples:

J-1.1.1 Samples shall be taken in a protected place which should not be exposed to atmospheric air.

J-1.1.2 The sampling instruments shall be clean and dry.

J-1.1.3 Samples, material being sampled, the sampling instruments and the containers for such samples shall be protected from adventitious contamination.

J-1.1.4 The sample shall be placed in clean and dry glass containers. The sample containers shall be of such a size that they are almost completely filled by the sample.

J-1.1.5 Each container shall be sealed air-tight after filling, and marked with full details of sampling, date of sampling batch or code number, name of manufacturer, and other important particulars of the consignment.

J-1.1.6 The samples shall be stored in such a manner that the temperature of the material does not vary markedly from ambient temperature. Samples should be protected from light.

J-2 SCALE OF SAMPLING

J-2.1 Lot

In a single consignment, all the packages containing material of the same grade and drawn from the same batch of manufacturer, shall constitute a lot. If the consignment consists of packages containing material of different grades or batch of manufacture, then the packages containing detergent of the same grade and batch of the manufacture shall be grouped together and each such group shall constitute a separate lot.

J-2.2 For ascertaining the conformity of the lot to the requirements prescribed in this standard, tests shall be carried out on each lot separately. The number (n) of packages to be selected for drawing samples shall depend upon the size (N) of the lot and shall be in accordance with Table 2.

J-2.3 The packages shall be selected at random. In order to ensure the randomness of selection, a random number table shall be used. For guidance and use of random number tables, IS 4905 may be used. In the absence of a random number table, the following procedure may be adopted:

‘Starting from any package in the lot, count them in one order as 1, 2, 3 up to r and so on where r is the integral part of N/n (N being the lot size and n the number of packages to be selected). Every r^{th} package thus counted shall be withdrawn to give the required sample size.

Table 2 Scale of Sampling

(Clause J-2.2)

Sl No.	No or Packages in the Lot (N)	No of Packages to be Selected (n)
(1)	(2)	(3)
i)	4 to 15	3
ii)	16 to 40	4
iii)	41 to 65	5
iv)	66 to 110	7
v)	111 and above	10

NOTE — When the size of the lot is 3 packages or less, the number of packages to be selected and the criteria for judging the conformity of the lot to the specification shall be as agreed between the interested parties.

J-3 PREPARATION OF GROSS SAMPLES, TEST SAMPLES AND REFEREE SAMPLES

J-3.1 Gross Samples

J-3.1.1.1 From each one of the packages as selected in J-2 draw one or more containers randomly. The material in the containers so chosen shall be nearly thrice the quantity required for the test as indicated in 7.2.

J-3.1.1.2 The material from the containers as selected in J-3.1.1 shall be mixed thoroughly to give the gross sample for the package.

J-3.2 Test Samples

J-3.2.1 Segregate carefully the gross samples (see I-3.1.1.1) of powders. From the gross sample, take a small but equal quantity of material and mix it thoroughly into a composite sample which should be of a size sufficient to carry out triplicate testing for all the characteristics specified under 7.2. The composite sample shall be divided into three equal parts; one for the purchaser, another for the supplier, and the third for the referee.

J-3.2.2 The remaining portion of the material in each one of gross samples shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the ‘n’ selected packages shall be for the purchaser, another for the supplier, and the third for the referee.

J-3.2.3 All the composite and individual samples shall be transferred to separate containers. These containers shall be air-tightly sealed with stoppers, and labelled with full particulars of identification given in **J-1.1.5**.

J-3.3 Referee Samples

J-3.3.1 The referee samples shall consist of a composite sample and a set of 'n' individual samples. All the

containers shall bear the seals of interested parties and shall be kept at a place agreed to between the two parties.

J-3.3.2 Referee samples shall be used in case of any dispute between the purchaser and the supplier.

ANNEX M

(Foreword)

COMMITTEE COMPOSITION

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE DEVELOPMENT OF THIS STANDARD

Soaps and Other Surface Active Agents Sectional Committee, CHD 25

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